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Adhesive bond primers are critical to the quality and performance of an adhesive system. Primers are used to enhance adhesion and to prevent corrosion of the metal surface. Most current primers do not meet the 240g/L compliance limit for volatile organic compounds (VOC's) and contain chromates. Waterborne primers, with reduced VOC levels, have been evaluated for future use as a replacement for these noncompliant primers. One primer being evaluated, BR®6757, is both a zero VOC and zero chromate primer.
The object of this study is to compare the performance of the conventional solvent based primers (BR®127 Cytec and EA®9205 Dexter) with the recently developed and commercially qualified waterborne primers (BR®6757 Cytec, BR®6747 Cytec, BR®6747-1 Cytec, EA®9257 Dexter), when coupled with various epoxy adhesives that are frequently used on naval aircraft. The mechanical and qualitative properties of these adhesive systems were evaluated via floating roller peel (FRP), single lap sheer (SLS), and wedge crack extension (WCE) tests.

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PERFORMANCE OF SOLVENT AND WATER BASED PRIMERS IN EPOXY ADHESIVE SYSTEMS

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Adhesive bond primers are critical to the quality and performance of an adhesive system. Primers are used to enhance adhesion and to prevent corrosion of the metal surface. Most current primers do not meet the 240g/L compliance limit for volatile organic compounds (VOC's) and contain chromates. Waterborne primers, with reduced VOC levels, have been evaluated for future use as a replacement for these non-compliant primers. One primer being evaluated, BR®6757, is both a zero VOC and zero chromate primer.

The objective of this study is to compare the performance of the conventional solvent based primers (BR®127 Cytec and EA®9205 Dexter) with the recently developed and commercially qualified waterborne primers (BR®6757 Cytec, BR®6747 Cytec, BR®6747-1 Cytec, EA®9257 Dexter), when coupled with various epoxy adhesives that are frequently used on naval aircraft. The mechanical and qualitative properties of these adhesive systems were evaluated via floating roller peel (FRP), single lap shear (SLS), and wedge crack extension (WCE) tests.

KEY WORDS: Adhesive Bonding, Primers, Volatile Organic Compounds

1. INTRODUCTION

The objective of this study is to compare the performance of the conventional solvent based primers (BR®127 Cytec and EA®9205 Dexter) with the recently developed and commercially qualified waterborne primers (BR®6757 Cytec, BR®6747 Cytec, BR®6747-1 Cytec, EA®9257 Dexter), when coupled with various epoxy adhesives that are frequently used on naval aircraft. Adhesive bonding primers are widely used in the aerospace industry to optimize the service life of the bonded component by protecting the newly pretreated surface from contamination, improving the overall performance of the component, and enhancing the environmental resistance of the bond interface [1, 2, 3, 4]. Currently qualified primers contain high levels of volatile organic compounds (VOC's) which are released into the atmosphere during application. These primers also contain chromium compounds as inhibitors. New environmental regulations require a reduction in the maximum allowable content of VOC's in adhesive primers.

As a result, water based primers have been developed that have reduced levels of VOC's and decreased levels of chromium compounds. One primer, Cytec's BR®6757, is both a zero VOC and zero chromate primer. Promising results have been seen in several studies conducted on commercially available water borne primers [3, 5-9]. The newer systems have also shown extreme sensitivity to primer layer thickness due to their inherently brittle nature [9]. Therefore, the thickness of the primer layer must be closely controlled to ensure optimum effectiveness.

2. EXPERIMENTAL

2.1 Materials 2024-T3 bare aluminum was used in this study. The aluminum was processed in accordance with ASTM Preparation of Aluminum Surfaces for Structural Adhesive Bonding (Phosphoric Acid Anodizing) (D-3933). A group of six adhesives coupled with three primers make up the matrix for the 121 °C (250 °F) cure systems. Similarly, a group of two adhesives coupled with four primers compose the matrix for the 177 °C (350 °F) systems.

TABLE 1 PRIMERS USED IN STUDY

MANUFACTURER	PRIMER	CARRIER	ADHESIVE CURE TEMP.
Cytec	BR®127	Solvent	121 °C
Cytec	BR®6747-1	Water	121 °C
Cytec	BR®6757	Water (Non-Chromated)	121 and 177 °C
Dexter	EA®9205	Solvent	177 °C
Dexter	EA®9257	Water	177 °C
Cytec	BR®6747	Water	177 °C

TABLE 2 ADHESIVES USED IN STUDY

MANUFACTURER	ADHESIVE	TYPE	CURE TEMP.
Cytec	FM®73	Film	121 °C
Cytec	FM®300-2K	Film	121 °C
Dexter	EA®9628	Film	121 °C
Cytec	FM®94M	Film	121 °C
Cytec	FM®300	Film	177 °C
Dexter	EA®9689	Film	177 °C

Tables 1 and 2, respectively, show the lists of the primers and the adhesives that were used in this study.

2.2 Bonding Procedure The same bonding procedure was used for all of the adhesive systems. Panels were bonded in a small vacuum/pressure tool assembly. The tool assembly was clamped between heated press platens during the bonding cycle. Bonding procedures were performed in accordance with manufacturer's instruction's with a heat up rate of 1.67 °C (3.0 °F) per minute and a cure time of 1 hour at either 121 °C (250 °F) or 177 °C (350 °F).

2.3 Test Methods

2.3.1 Wedge Crack Extension Tests Panels with dimensions of 15.24 x 15.24 x 0.317 cm were bonded for each primer system. Testing was performed on the 2.54 cm wide specimens in accordance with ASTM Adhesive-Bonded Surface Durability of Aluminum (Wedge Crack Extension Test) (D-3762). Readings were taken after exposures of 0, 1, 4, 24, 168, and 672 hours in a humidity chamber at 100 % relative humidity and 60 °C.

2.3.2 Lap Shear Tensile Tests Panels with five 2.54 cm wide fingers were bonded together for each primer system. There was a 0.157 cm bonded overlap for each set of bonded panels. Testing was performed on the 2.54 cm wide specimens in accordance with ASTM Strength and properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (D-1002). The test conditions were (1) ambient temperature, (2) 104 °C after 2160 hours of exposure to 60 °C and 100% relative humidity, (3) ambient temperature after 2160 hours of exposure to 60 °C and 100% relative humidity followed by drying in a desicator for 336 hours, (4) –55 °C.

2.3.3 Floating Roller Peel Tests One panel with dimensions 15.24 x 27.94 x .0635 cm and another panel of dimensions 15.24 x 25.40 x 0.160 cm were bonded together for each primer system. Testing was performed on the 2.54 cm wide specimens in accordance with ASTM Floating Roller Peel Resistance of Adhesives (D-3167). Test conditions were (1) ambient temperature, (2) ambient temperature with deionized water squirted into fail area during pull, (3) 104 °C after 2160 hours of exposure to 60 °C and 100% relative humidity, (4) ambient temperature after 2160 hours of exposure to 60 °C and 100% relative humidity followed by drying in a desicator for 336 hours, (5) –55 °C.

3. RESULTS AND DISCUSSION

3.1 FM®73 Film Adhesive

3.1.1 Wedge Crack Extension Tests The wedge crack extension test growth profiles for the FM®73 film adhesive systems are shown in figure 1. All of the specimens showed stable crack growth that did not accelerate over time. The average total crack lengths for specimens at the 24-hour mark were 0.575, 0.536, and 0.584 cm for those primed with BR®127, BR®6747-1, and BR®6757, respectively. All three of the systems performed well with less than .635 cm (0.25 in.) of crack growth over the first 24-hour period. The systems exhibited an average of 100% cohesive failure for all three primer systems.

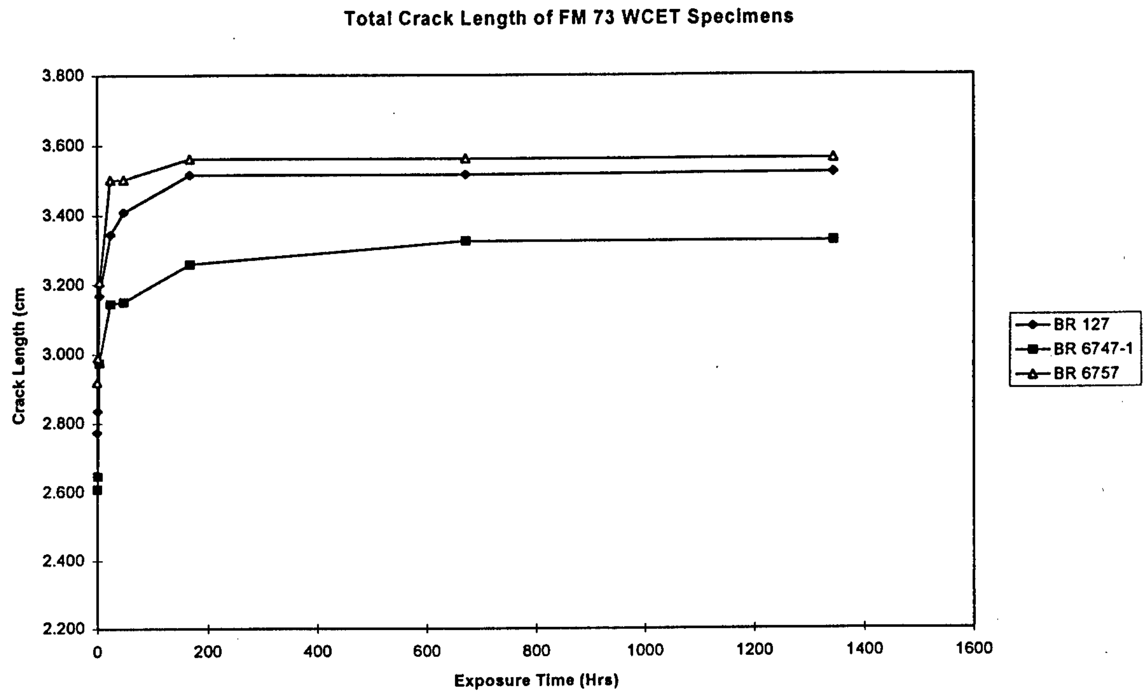

FIGURE 1. Growth profiles for wedge crack extension test of FM®73 film adhesive. Values are in cm.

3.1.2 Lap Shear Tensile Tests The lap shear results for FM®73 are given in table 3; the values are in MPa with psi values given in parentheses. Specimens primed with the water based primers BR®6747-1 and BR®6757 showed equal to higher performance than the solvent based BR®127 at all test conditions. Specimen failure modes for all three systems were 75-100% cohesive for ambient temperature and exposed/104 °C conditions. BR®127 systems also showed a high percentage of cohesive failure for exposed/dry and –55 °C test conditions while the water based primers showed a lower 25-75% cohesive failure mode at the same conditions.

3.1.3 Floating Roller Peel Tests The results of the floating roller peel tests for FM®73 are given in table 4; the values are in MPa with psi values in parentheses. The peel strengths of the water based BR®6747-1 specimens compared well with the BR®127 specimens, with the exception of the –55 °C condition. Likewise, the performance of BR®6757 specimens was equivalent or better than BR®127 at all conditions except exposed/dry. The failure modes of all three systems showed mostly cohesive failure (50-100%) at all conditions, except for the previously noted specimens which showed more adhesive character (about 75% adhesive failure).

TABLE 2 LAP SHEAR TEST RESULTS FOR FM®73 FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	41.46 MPa (6014.40 psi)	42.19 MPa (6119.70 psi)	41.59 MPa (6032.30 psi)
60°C/100%RH exp. then 104°C	3.83 MPa (556.00 psi)	4.01 MPa (581.50psi)	3.97 MPa (576.40 psi)
60°C/100%RH exp. then ambient temp.	35.28 MPa (5117.70 psi)	38.42 MPa (5572.10 psi)	36.61 MPa (5309.40 psi)
-55 °C	47.22 MPa (6848.40 psi)	42.95 MPa (6228.80 psi)	47.22 MPa (6848.60 psi)

TABLE 3 ROLLER PEEL TEST RESULTS FOR FM®73 FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	0.55 MPa (80.12 psi)	0.58 MPa (84.24 psi)	0.52 MPa (74.86 psi)
Ambient Temp. w/ DI squirt	0.53 MPa (76.40 psi)	0.58 MPa (84.88 psi)	0.52 MPa (75.92 psi)
60°C/100%RH exp. then 104°C	0.16 MPa (22.73 psi)	0.16 MPa (22.81 psi)	0.20 MPa (28.64 psi)
60°C/100%RH exp. then ambient temp.	0.53 MPa (77.40 psi)	0.56 MPa (81.86 psi)	0.25 MPa (36.50 psi)
-55 °C	0.38 MPa (54.75 psi)	0.18 MPa (26.62 psi)	0.35 MPa (51.25 psi)

3.2 FM®300-2K Film Adhesive

3.2.1 Wedge Crack Extension Tests The wedge crack extension test growth profiles for the FM®300-2K film adhesive systems are shown in figure 2. All of the specimens showed a stable growth pattern and did not accelerate over time. All specimens grew less than 0.635 cm (0.25 in.) in the first 24 hours. The specimens showed crack growths of 0.025 cm (0.010 in.), 0.033 cm (0.013 in.), and 0.081 cm (0.032 in.) for BR®127, BR®6747-1, and BR®6757 respectively. Specimens showed average cohesive failure of 90%, 100%, and 100% for BR®127, BR®6747-1, and BR®6757 respectively. Specimens primed with BR®6747-1 showed the smallest total crack length having a crack length of 4.153 cm (1.635 in.) at 1344 hours of exposure.

3.2.2 Lap Shear Tensile Tests The lap shear results for FM®300-2K are given in table 5; the values are in MPa with psi values in parentheses. The BR®6747-1 specimens performed the best of the three systems at all test conditions. The water based non-chromated BR®6757 primed specimens exhibited comparable but slightly lower shear strength values than the solvent based BR®127 specimens. The specimens at most test conditions averaged approximately 75% cohesive failure for all systems. The exceptions being BR®6757/FM®300-2K at exposed/104 °C conditions and the BR®6747-1/FM®300-2K at exposed/dry conditions which both exhibited predominantly adhesive failure modes (average 10% cohesive). All samples at –55 °C also exhibited an average of 10% cohesive failure. This is difficult to explain since a predominantly adhesive failure would imply a decrease in strength, but this is not the case here.

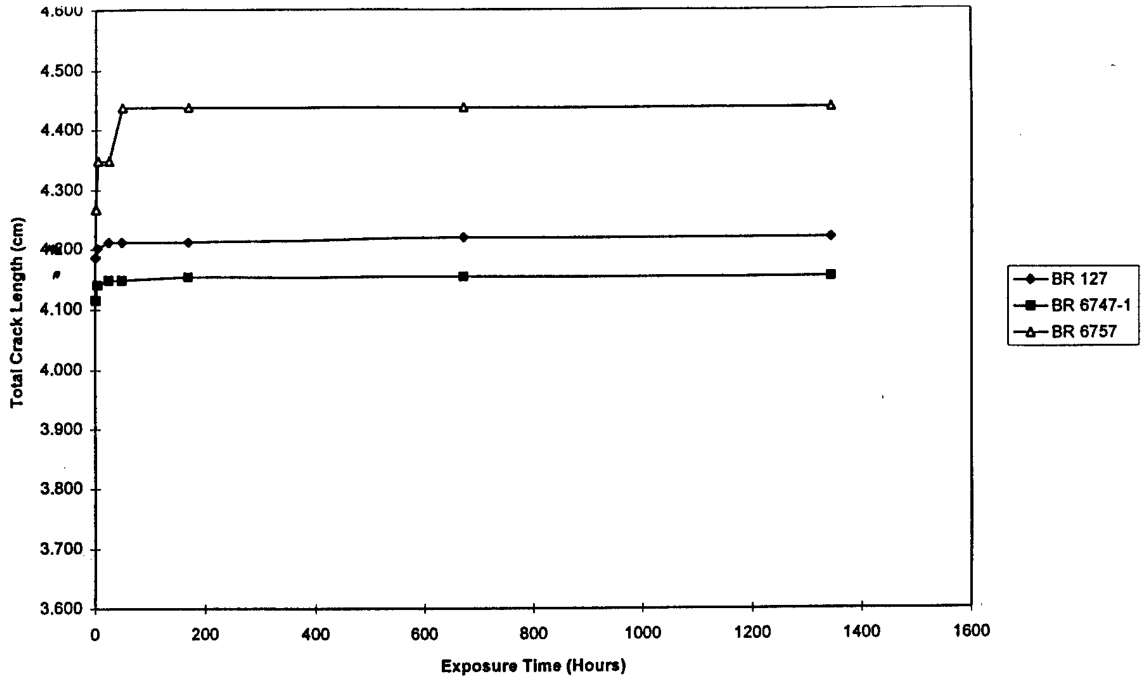

FIGURE 2. Growth profiles for wedge crack extension test of FM®300-2K film adhesive. Values are in cm .

TABLE 5 LAP SHEAR RESULTS FOR FM®300-2K FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	40.13 MPa (5821.10 psi)	41.10 MPa (5961.30 psi)	36.89 MPa (5350.10 psi)
60°C/100%RH exp. then 104°C	19.72 MPa (2859.60 psi)	21.74 MPa (3153.80 psi)	15.20 MPa (2204.60 psi)
60°C/100%RH exp. then ambient temp.	38.70 MPa (5613.70 psi)	40.95 MPa (5939.50 psi)	34.27 MPa (4970.30 psi)
-55 °C	27.50 MPa (3988.40 psi)	31.18 MPa (4521.70 psi)	26.27 MPa (3810.40 psi)

TABLE 6 ROLLER PEEL RESULTS FOR FM®300-2K FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	0.25 MPa (36.85 psi)	0.18 MPa (26.24 psi)	0.25 MPa (36.83 psi)
Ambient Temp. w/ DI squirt	0.24 MPa (34.60 psi)	0.19 MPa (27.83 psi)	0.25 MPa (35.63 psi)
60°C/100%RH exp. then 104°C	0.28 MPa* (40.33 psi)*	0.22 MPa (31.97 psi)	0.25 MPa (36.28 psi)
60°C/100%RH exp. then ambient temp.	0.23 MPa (33.37 psi)	0.19 MPa** (27.34 psi)**	0.21 MPa (30.37 psi)
-55 °C	0.06 MPa (8.22 psi)	0.04 MPa (6.26 psi)	0.11 MPa (16.60 psi)

*EXPOSED 83 DAYS
**EXPOSED 99 DAYS

3.2.3 Floating Roller Peel Tests The results of the floating roller peel resistance testing for FM®300-2K are given in table 6; values are in MPa with psi in parentheses. The water based non-chromated BR®6757 performed equivalently to the BR®127 for all specimens at all conditions. The specimens primed with BR®6747-1 exhibited peel

of the all the specimens at exposed/104 °C conditions averaged 75-100% cohesive. At all of the other conditions a lower percentage of cohesive failure was exhibited, 10-50%.

3.3 EA®9628 Film Adhesive

3.3.1 Wedge Crack Extension Tests The wedge crack extension test growth profiles for the EA®9628 film adhesive systems are shown in figure 3. All three primer systems worked well with less than 0.635 cm (0.25 in.) over the first 24 hours of exposure. Specimens primed with water borne BR®6747-1 had the lowest average total crack growth over the entire 1344 hours. The average total crack length was 0.025 cm (0.03 in.), .046 cm (0.018 in.), and .038 cm (0.015 in.) for specimens primed with BR®127, BR®6747-1 and BR®6757 respectively. All of the specimens exhibited an average of 100% cohesive failure.

3.3.2 Lap Shear Tensile Tests The lap shear results for EA®9628 are given in table 7 the values are in MPa with psi in parentheses. The water based BR®6747-1 and BR®6757 primed specimens exhibited equal or slightly higher shear strengths than BR®127 for all test conditions, with one exception. BR®6747-1/EA®9628 at exposed/dry conditions showed a 50% reduction of shear strength compared to that of the BR®6757 system with the same conditions. All specimens at all test conditions exhibited 75-100% cohesive failure.

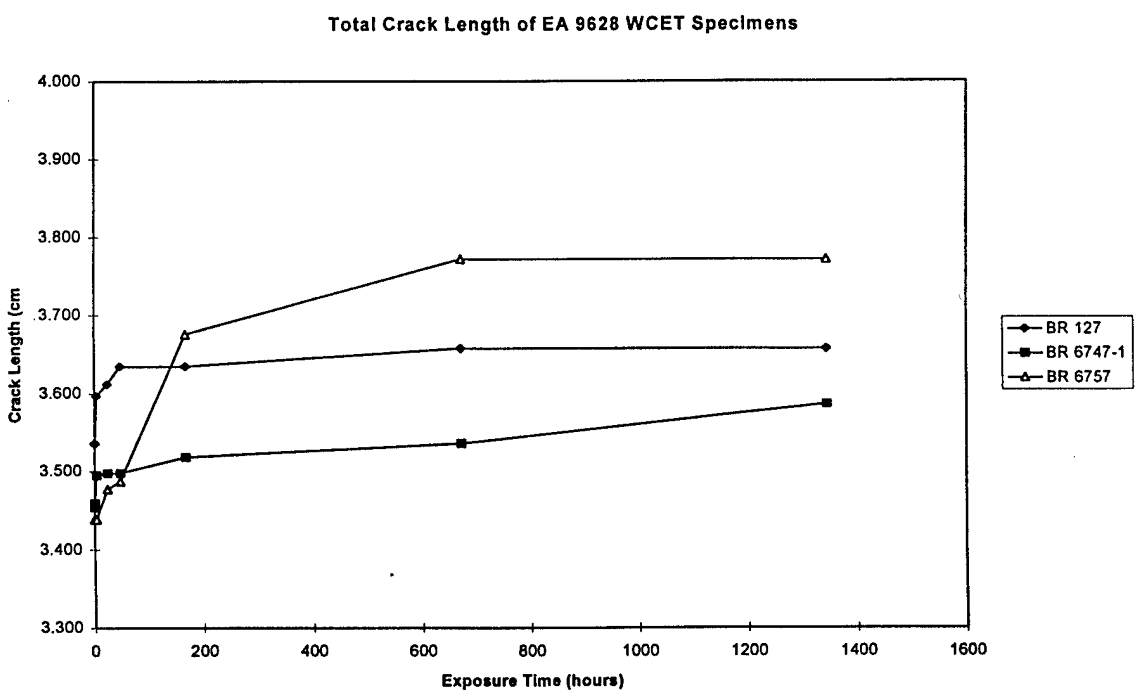

FIGURE 3. Growth profiles for wedge crack extension test of EA®9628 film adhesive. Values are in cm

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	39.24 MPa (5690.90 psi)	45.90 MPa (6657.09 psi)	47.25 MPa (6582.50 psi)
60°C/100%RH exp. then 104°C	4.63 MPa (672.20 psi)	6.47 MPa (939.00 psi)	5.41 MPa (785.10 psi)
60°C/100%RH exp. then ambient temp.	32.95 MPa (4778.80 psi)	18.29 MPa (2652.80 psi)	37.02 MPa (5369.60 psi)
-55 °C	41.79 MPa (6061.63 psi)	45.73 MPa (6632.20 psi)	46.76 MPa (6782.50 psi)

TABLE 8 ROLLER PEEL RESULTS FOR EA®9628 FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	0.36 MPa (52.41 psi)	0.39 MPa (56.02 psi)	0.39 MPa (56.26 psi)
Ambient Temp. w/ DI squirt	0.36 MPa (52.88 psi)	0.39 MPa (56.44 psi)	0.37 MPa (54.29 psi)
60°C/100%RH exp. then 104°C	0.42 MPa (60.95 psi)	0.45 MPa (65.83 psi)	0.44 MPa (64.51 psi)
60°C/100%RH exp. then ambient temp.	0.40 MPa (57.83 psi)	0.42 MPa (60.94 psi)	0.39 MPa (56.53 psi)
-55 °C	0.27 MPa (39.30 psi)	0.16 MPa (23.00 psi)	0.32 MPa (46.85 psi)

3.3.3 Floating Roller Peel Tests Floating roller peel test results for EA®9628 are given in table 8; values are in MPa with psi in parentheses. The peel strength data for all specimens show that there is virtually no difference between the water based and the solvent based primer systems for all testing conditions with a lone exception. BR®6747-1/EA®9628 at –55 °C exhibits a 50% decrease in peel strength to that of the other water based primer BR®6757. All specimens at all test conditions, other than –55 °C, showed 75-100% cohesive failure. Specimens for all three primer systems at –55 °C showed an average of 25% cohesive failure.

3.4 FM®94M Film Adhesive

3.4.1 Wedge Crack Extension Tests Wedge crack extension test growth profiles for FM®94M are shown in figure 5. All three primer systems performed well with stable growth patterns and less than 0.635 cm (0.25 in.) in the first 24 hours of exposure. All specimens averaged 100% cohesive failure

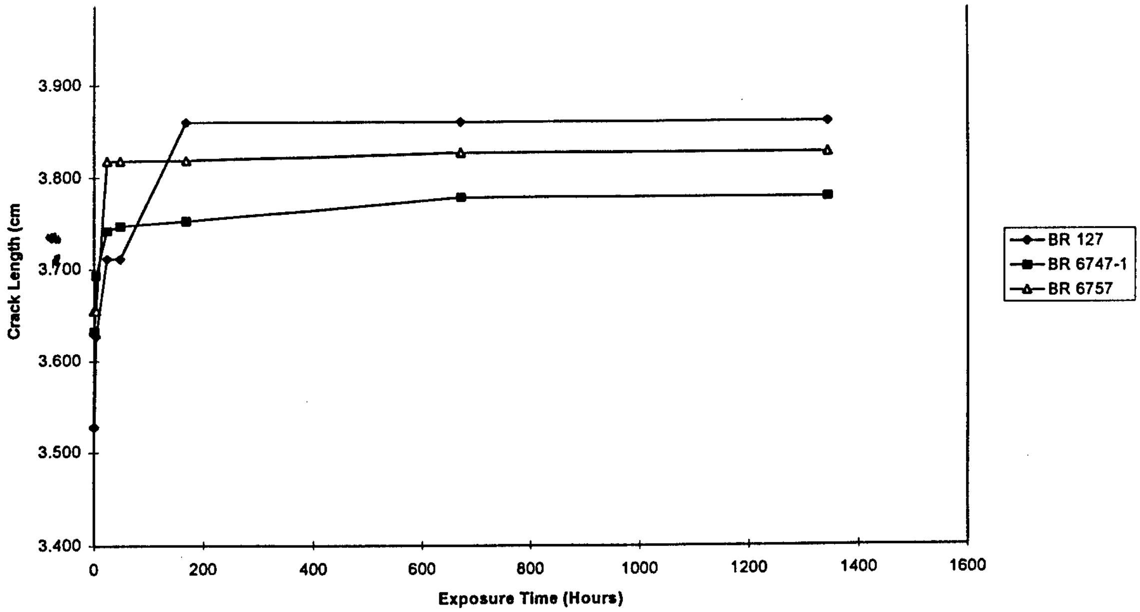

FIGURE 5 Growth profiles of wedge crack extension tests for FM®94M film adhesive. Values are in cm.

3.4.2 Single Lap Shear Tension Tests The single lap shear tension test results are given in table 9; values are in MPa with psi in parentheses.

TABLE 9 LAP SHEAR RESULTS FOR FM®94M FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	38.55 MPa (5591.40 psi)	40.16 MPa (5825.20 psi)	38.09 MPa (5525.10 psi)
60°C/100%RH exp. then 104°C	9.17 MPa (1330.50 psi)	93.37 MPa (1354.20 psi)	83.58 MPa (1212.30 psi)
60°C/100%RH exp. then ambient temp.	34.43 MPa (4993.80 psi)	36.17 MPa (5246.50 psi)	32.26 MPa (4679.40 psi)
-55 °C	38.22 MPa (5544.00 psi)	40.65 MPa (5895.50 psi)	38.61 MPa (5600.60 psi)

TABLE 10 ROLLER PEEL RESULTS FOR FM®94M FILM ADHESIVE

	BR®127	BR®6747-1	BR®6757
Ambient Temp.	0.43 MPa (62.64 psi)	0.44 MPa (63.72 psi)	0.43 MPa (62.15 psi)
Ambient Temp. w/ DI squirt	0.40 MPa (58.49 psi)	0.43 MPa (62.64 psi)	0.43 MPa (63.02 psi)
60°C/100%RH exp. then 104°C	0.39 MPa* (56.48 psi)*	0.40 MPa (57.88 psi)	0.35 MPa (51.38 psi)
60°C/100%RH exp. then ambient temp.	0.43 MPa (62.26 psi)	0.43 MPa** (63.04 psi)**	0.21 MPa (30.76 psi)
-55 °C	0.31 MPa (44.60 psi)	0.25 MPa (36.36 psi)	0.25 MPa (37.19 psi)

The shear strength of specimens primed with BR®6747-1 and BR®6757 were equivalent or better than the strength of the specimens primed with BR®127 at all test conditions. Failure modes for all specimens were cohesive in character, an average of 75-100% cohesive failure was observed.

3.4.3 Floating Roller Peel Tests The floating roller peel test results for FM®94M are given in table 10; values are in MPa with psi in parentheses. Data collected from all three systems show that there is virtually no difference between the water based and solvent based primers at any test condition. One exception, are the BR®6757 specimens at exposed/dry conditions. There was approximately a 50% reduction in peel strength when compared to the other primers at this condition. Failure modes were mostly cohesive in nature for all specimens. An average of 75-100% cohesive failure was typically observed.

3.5 FM®300K Film Adhesive

3.5.1 Wedge Crack Extension Tests Wedge crack extension test growth profiles for FM®300K are shown in figure 5. All specimens exhibited a stable crack growth during the testing. The water based EA®9257 and BR®6757 primed specimens showed similar crack lengths to that of the solvent based EA®9205. All three had less than 0.635 cm (0.25 in.) growth in the first 24 hours of exposure. Water based BR®6747, however, had a much larger total crack length than did the other primer systems. Specimens exhibited and average of 75%, 100%, 100%, and 100% cohesive failure for EA®9205, EA®9257, BR®6747, and BR®6757 respectively.

3.5.2 Single Lap Shear Tension Tests Single lap shear tension test results are given in table 11; values are in MPa with psi in parentheses. All four primer systems performed equivalently at all test conditions. There was no significant difference observed in the results of the water based verses the solvent based systems. Failure modes of the

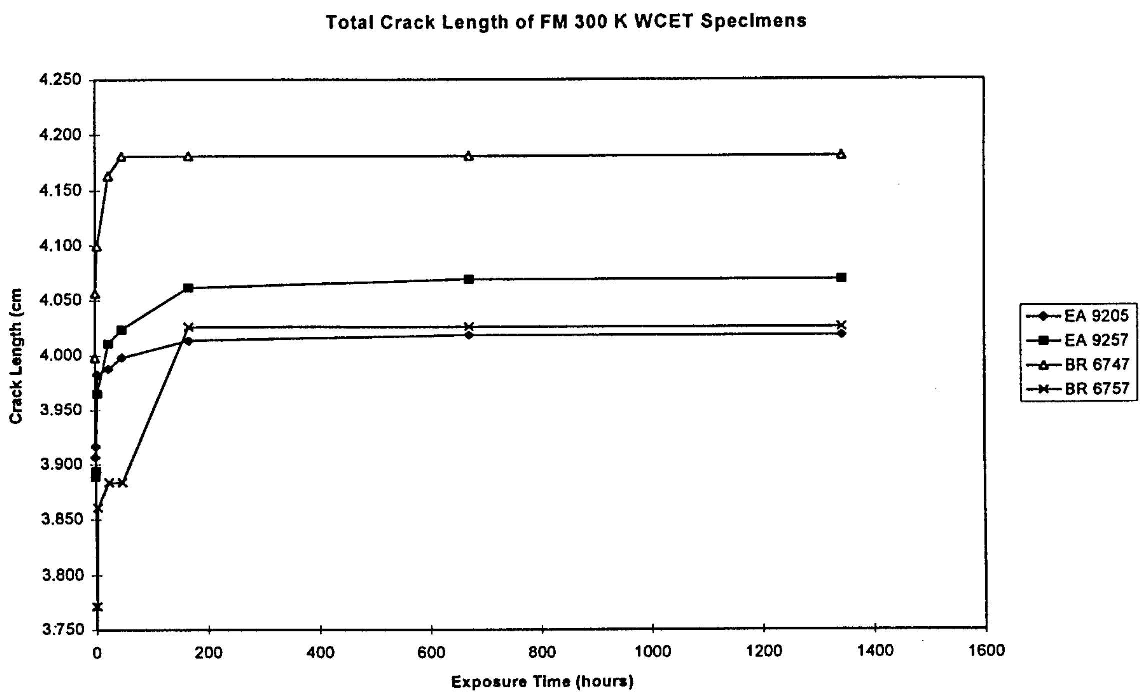

FIGURE 5 Growth profile of wedge crack extension tests for FM®300K film adhesive. Values are in cm.

specimens exhibited a significant amount of both adhesive and cohesive failure. An average of 25-75% cohesive failure was observed for specimens at all test conditions except –55 °C. Specimens pulled at -55 °C showed a high percentage of adhesive failure, an average of 75-90%.

3.5.3 Floating Roller Peel Tests Floating roller peel test results are given in table 12; values are in MPa with psi in parentheses. The water based EA®9257 performed better than any of the three other primer systems. The solvent based primer, EA®9205, gave the poorest results. Failure modes for the specimens at most test conditions showed about equal amounts of cohesive and adhesive failure. Specimens pulled at -55 °C, exhibited a higher average of adhesive failure, 75-90%.

TABLE 11 LAP SHEAR RESULTS FOR FM®300K FILM ADHESIVE

	EA®9205	EA®9257	BR®6747	BR®6757
Ambient Temp.	33.98 MPa (4928.30 psi)	34.92 MPa (5065.20 psi)	35.22 MPa (5108.70 psi)	32.52 MPa (4716.90 psi)
60°C/100%RH exp. then 104°C	20.06 MPa (2910.20 psi)	16.06 MPa (2329.20 psi)	19.74 MPa (2863.80 psi)	18.26 MPa (2648.20 psi)
60°C/100%RH exp. then ambient temp.	35.12 MPa (5094.40 psi)	35.02 MPa (5078.60 psi)	35.03 MPa (5081.20 psi)	34.98 MPa (5073.60 psi)
-55 °C	28.92 MPa (4195.20 psi)	31.66 MPa (4591.20 psi)	33.29 MPa (4828.00 psi)	32.50 MPa (4714.50 psi)

TABLE 12 ROLLER PEEL RESULTS FOR FM®300K FILM ADHESIVE

	EA®9205	EA®9257	BR®6747	BR®6757
Ambient Temp.	0.05 MPa (7.41 psi)	0.28 MPa (40.60 psi)	0.20 MPa (29.13 psi)	0.16 MPa (23.53 psi)
Ambient Temp. w/ DI squirt	0.05 MPa (7.65 psi)	0.25 MPa (36.91 psi)	0.20 MPa (28.98 psi)	0.17 MPa (24.34 psi)
60°C/100%RH exp. then 104°C	0.27 MPa (38.95 psi)	0.32 MPa (46.20 psi)	0.29 MPa (42.66 psi)	0.31 MPa (45.44 psi)
60°C/100%RH exp. then ambient temp.	0.05 MPa (7.31 psi)	0.30 MPa (43.32 psi)	0.09 MPa (13.84 psi)	0.04 MPa (5.59 psi)
-55 °C	0.05 MPa (6.72 psi)	0.10 MPa (14.41 psi)	0.07 MPa (9.63 psi)	0.05 MPa (7.94 psi)

3.5 EA®9689 Film Adhesive

3.6.1 Wedge Crack Extension Tests Wedge crack extension test growth profiles for EA®9689 are shown in figure 6. Two of the water based primers, EA®9257 and BR®6757 performed as well or better than the solvent based EA®9205. The BR®6747 performed the worst among the primer systems, as was the case with the BR®6747/FM®300K system. Specimens exhibited an average of 100%, 100%, 25%, and 100% cohesive failure for EA®9205, EA®9257, BR®6747, and BR®6757 respectively.

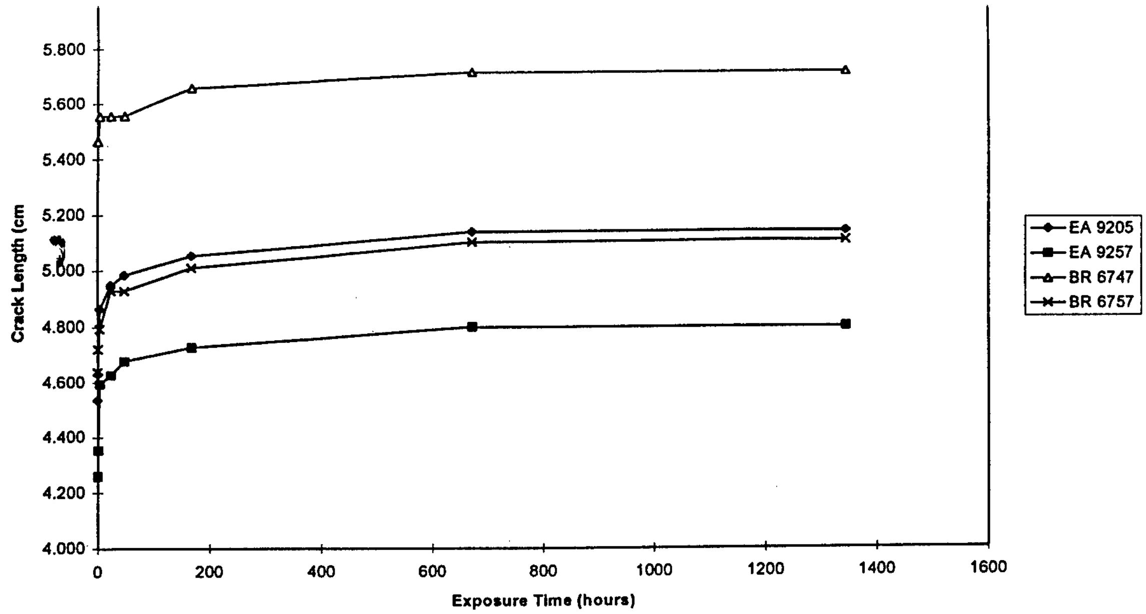

FIGURE 6 Growth profile of wedge crack extension tests of EA®9689 film adhesive. Values in cm.

3.6.2 Single Lap Shear Tension Tests Single lap shear tension test results are given in table 13; values are in MPa with psi in parentheses. All specimens tested for the four primer systems at all test conditions performed equally well. There was no significant difference between the performance of the water based primer specimens and the solvent based primer specimens. Failure modes varied from primer to primer and between different test conditions. Failure modes for the –55 °C pulls can be characterized as predominantly adhesive, an average of 75-100% adhesive failure. The specimens pulled at the other test conditions showed more cohesive failure, in most cases an average of 50-75% cohesive failure.

3.6.3 Floating Roller Peel Data Floating roller peel test results are given in table 14; values are in MPa with psi in parentheses. The water based BR®6757 and EA®9257 performed the best of all the primer systems. In comparison, the solvent based EA®9205 and water based BR®6747 performed poorly. Failure modes of all specimens can be characterized as mostly adhesive. The systems that had a greater peel strength, BR®6757 and EA®9257, showed some cohesive failure, an average of 25-50%, the other two primers were nearly 100% adhesive.

TABLE 13 LAP SHEAR RESULTS FOR EA®9689 FILM ADHESIVE

	EA®9205	EA®9257	BR®6747	BR®6757
Ambient Temp.	25.15 MPa (3647.70 psi)	26.13 MPa (3790.00 psi)	24.55 MPa (3561.20 psi)	25.74 MPa (3733.30 psi)
60°C/100%RH exp. then 104°C	18.74 MPa (2717.40 psi)	16.79 MPa (2435.50 psi)	18.73 MPa (2716.60 psi)	18.71 MPa (2713.60 psi)
60°C/100%RH exp. then ambient temp.	21.90 MPa (3175.80 psi)	20.60 MPa (2987.40 psi)	20.82 MPa (3020.40 psi)	21.78 MPa (3159.20 psi)
-55 °C	19.51 MPa (2830.10 psi)	25.74 MPa (3732.60 psi)	24.24 MPa (3515.30 psi)	26.41 MPa (3830.50 psi)

TABLE 14 ROLLER PEEL RESULTS FOR EA®9689 FILM ADHESIVE

	EA®9205	EA®9257	BR®6747	BR®6757
Ambient Temp.	0.04 MPa (5.42 psi)	0.11 MPa (16.65 psi)	0.04 MPa (5.21 psi)	0.12 MPa (17.39 psi)
Ambient Temp. w/ DI squirt	0.03 MPa (5.06 psi)	0.11 MPa (16.96 psi)	0.04 MPa (5.45 psi)	0.11 MPa (16.03 psi)
60°C/100%RH exp. then 104°C	0.04 MPa (6.00 psi)	0.08 MPa (11.83 psi)	0.03 MPa (4.78 psi)	0.10 MPa (14.26 psi)
60°C/100%RH exp. then ambient temp.	0.04 MPa (6.31 psi)	0.08 MPa** (11.68 psi)**	0.04 MPa (6.19 psi)	0.07 MPa (10.63 psi)
-55 °C	0.05 MPa (7.21 psi)	0.12 MPa (17.33 psi)	0.04 MPa (5.63 psi)	0.14 MPa (20.15 psi)

**EXPOSED 99 DAYS

4. SUMMARY

The water based primers, BR®6747-1 and BR®6757, when coupled with the 121 °C (250 °F) adhesives used in this study, generally performed well in comparison with the solvent based BR®127. The chromated water based primer, BR®6747-1, was the best performer throughout all of the testing. Specimens primed with the non chromated water based BR®6757 exhibit properties that are comparable to the solvent based BR®127 in single lap shear and floating roller peel testing. However, wedge crack extension test specimens primed with BR®6757 exhibited larger crack growth most noticeably when coupled with FM®300-2K and EA®9628 adhesives. This may be a cause for concern and require further testing and analysis. Another area of concern that may require future testing is the noticeable discrepancy in failure modes in a few cases. These discrepancies in failure modes may be a result of batch variance. Additional testing is also planned for test conditions were one primer/adhesive system shows a significant decrease in strength when compared to the others. Again, this may be caused by batch variance, or it may be evidence of a flaw in the ability of the primer/adhesive system to perform under certain conditions.

The 177 °C (350 °F) adhesives when coupled with the water based BR®6757 and EA®9257 were equivalent or better when compare to the solvent based EA®9205 specimens in WCET, SLST, and FRPT testing. However, the chromated water based BR®6747 specimens consistently performed poorly in the different tests. This is a puzzling result since the only major difference between the BR®6757, which performed well, and the BR®6747 is the presence of chromates and one would think that a chromated inhibitor would outperform a non-chromated one. Further testing with the BR®6747 may be necessary or testing with the newer formulation, BR®6747-1, with the 177 °C (350 °F) adhesives may resolve this concern.

Overall, the water based systems evaluated have shown great promise for the replacement of solvent based systems.

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